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PROVISIONAL SPECIFICATION

Applicant:

GBC SCIENTIFIC EQUIPMENT PTY LTD

A.C.N. 005 472 686

Invention Title:

RHEOMETER

The invention is described in the following statement:

RHEOMETER

Field of the Invention

This invention relates to a rheometer and, in particular, to a rheometer which can measure the complex viscosity and complex modulus of small volumes of fluids, as well as other rheological properties of materials such as elasticity.

Background Art

Most conventional rheometers which measure viscosity, often simply referred to as viscometers, cannot be used for measuring viscosities of non-Newtonian samples. Furthermore, most conventional viscometers need large amounts of sample in order to enable measurements to be made. This is therefore a significant disadvantage, because in many cases, only very small amounts of sample are available for analysis.

Summary of the Invention

The object of the present invention is to provide a rheometer which can provide measurements with only small amounts of sample and which can also accurate measurements.

The invention may be said to reside in a rheometer for determining a rheological property of a sample, including:

vibrating means for applying an alternating movement to a surface of the sample for causing an alternating movement of the sample;

force measuring means for providing a force signal indicative of the reaction force exerted by the sample on the vibrating means;

displacement measuring means for providing a signal indicative of the alternating movement of the sample;

processing means for receiving the force signal

and the movement signal to determine the rheological property of the sample; and

signal generating means for supplying to the vibrating means a frequency sweep signal having a monotonic group delay function to cause the vibrating means to supply the alternating movement of the sample.

Thus, this aspect of the invention enables very small amounts of sample to be used to enable measurements to take place, and also the generating means ensures that harmonics from non-linearities (ie. distortion) are distributed in a well-defined way, rather than randomly across the frequency spectrum, which can be corrected for in the data processing by the processing means. Furthermore, the effect of intermittent external noise sources is confined to the frequencies during which they occurred, rather than being spread across the whole spectrum. Thus, the nature of the signal applied to the vibrating means which creates the reaction force, and therefore produces the force signal and the movement signal, enables better processing and therefore more accurate results to be obtained.

Preferably, the frequency sweep signal has a monotonic group delay function with a maximum value less than the acquisition period.

Preferably the frequency sweep signal has small crest factors and most preferably, close to 3dB if using a flat amplitude envelope in the time domain. This provides the best practical crest factor and so gives the highest signal to noise ratio in terms of analogue to digital resolution and noise generated by the sampling electronics. The frequency sweep function also provides the ability to prescribe the amplitude envelope in the time domain and thus gives the rheologist control of this parameter. This in turn enables the maximum strain rates

to be constrained to within the linear region of the sample.

The frequency sweep signal also enables fading of the start and end points to zero amplitude, which means that there are no unexpected transitions in the signal being injected into the sample, and so the sample integrity is preserved.

This form of signal also provides the ability to prescribe the spectral content envelope in the frequency domain, such as flat linear and log envelopes. This enables the rheologist to control how much energy is put into the sample at different frequencies. It also enables the signal to noise ratio of the stress measurement to be equalised, since typical samples do not have a flat transfer function.

Preferably the vibrating means includes a driver having terfenite material and means for supplying an electromagnetic force to the terfenite material to produce the alternating movement.

Preferably the apparatus includes a sample support comprised of a top plate and a bottom plate which define a space for receiving the sample.

Preferably the displacement measuring means comprises a displacement transducer.

Preferably the force measuring means comprises a load cell.

Preferably the processing means includes an analogue to digital converter for converting the signal from the load cell to a digital signal, and an analogue to digital converter for converting the signal from the displacement

measuring means to a digital signal.

Preferably the processor is for determining the fourier transform of both the force signal and the movement signal, and the ratio of the fourier transform of the force signal $F(\omega)$ to the fourier transform of the movement signal $H(\omega)$.

Preferably at least one of the top plate and bottom plate is circular and has a radius a and the plates are separated by an average distance h and the property which is calculated is the complex modulus

$$G^*(\omega) = h^3/3\pi a^4 \times F(\omega)/H(\omega).$$

The invention may be said to reside in a method of determining a rheological property of a sample, including:

applying by a vibrating means an alternating movement to a surface of the sample for causing an alternating movement of the sample;

measuring a force signal indicative of a reaction force exerted by the sample;

measuring a signal indicative of the alternating movement of the sample;

processing the force signal and the movement signal to determine the rheological property of the sample; and

supplying to the vibrating means a frequency sweep signal having a monotonic group delay function to produce the alternating movement of the sample.

Preferably, the frequency sweep signal has a monotonic group delay function with a maximum value less than the acquisition period.

Preferably the frequency sweep signal has small crest factors and most preferably, close to 3dB if using a flat

amplitude envelope in the time domain.

Preferably the vibrating means includes a driver including terfenite material and means for supplying an electromagnetic force to the terfenite to produce the alternating movement.

Preferably the method includes supporting the sample between a top plate and a bottom plate which define a space for receiving the sample.

Preferably the displacement is measured by a displacement transducer.

Preferably the force is measured by a load cell.

Preferably the processing includes converting the force signal to a digital signal, and converting the displacement signal to a digital signal.

Preferably the processor further includes determining the fourier transform of both the force signal and the movement signal, and the ratio of the fourier transform of the force signal to the fourier transform of the movement signal.

Preferably at least one of the top plate and bottom plate is circular and has a radius a and the plates are separated by an average distance h and the property which is calculated is the complex modulus

$$G^{*}(\omega) = h^{3}/3\pi a^{4} \times F(\omega)/H(\omega).$$

A further aspect of the invention is concerned with problems which are created when a fluid sample is located between the top plate and the bottom plate of the rheometer. If the sample is in the form of a fluid, the

meniscus of the fluid can effectively form a spring between the top and bottom plates. That is, when the alternating force is applied to the sample, the meniscus effectively provides a resistance or load against that force, and this in turn causes errors in the resulting measurements.

The second aspect of the present invention overcomes this problem.

Thus, the second aspect of the invention may be said to reside in a rheometer for determining a rheological property of a fluid sample, including:

vibrating means for applying an alternating movement to a surface of the sample for causing an alternating movement of the sample;

force measuring means for providing a force signal indicative of the reaction force exerted by the sample on the vibrating means;

displacement measuring means for providing a signal indicative of the alternating movement of the sample;

processing means for receiving the force signal and the movement signal to determine the rheological property of the sample; and

sample support means having a top plate and a bottom plate between which a space is provided for receiving the sample, one of said plates being moveable relative to the other plate by the vibrating means, said one of said plates having a side edge, means for causing the fluid sample to extend up the side wall of the said one of the plates to form a concave meniscus so that upon movement of the said one of the plates, the meniscus will slip on the edge of the top plate thereby reducing the spring nature of the meniscus to reduce errors in the resulting measurement due to the spring nature of the meniscus.

Preferably the said means comprises a quartz surface on the at least said one plate at least in the vicinity of the meniscus so that the fluid flows up the said side surface thereby creating the meniscus which extends up the side surface of the said one plate.

Preferably the said at least one plate is formed from steel and the quartz surface is formed by vacuum depositing quartz onto the said steel plate.

Preferably the quartz surface has a thickness of about 100 micrometers.

Preferably both the top plate and the bottom plate are provided with the quartz surface having the thickness of about 100 micrometers.

This embodiment, in relation to aqueous fluids, lowers the contact angle of the meniscus with the said one plate and allows the fluid sample to thereby be used with steel plates. This in turn allows relatively cheap plates to be used and also plates which are easy to clean and less porous than plain hardened stainless steel plates.

Preferably the said one of the plates comprises the top plate.

Thus, the second aspect of the invention may be said to reside in a method of determining a rheological property of a sample fluid, including:

applying by way of a vibrating means an alternating movement to a surface of the sample for causing an alternating movement of the sample;

measuring a force signal indicative of the reaction force exerted by the sample on the vibrating means;

measuring a signal indicative of the alternating movement of the sample;

processing the force signal and the movement signal to determine the rheological property of the sample; and

supporting the sample between a top plate and a bottom plate between which a space is provided for receiving the sample fluid, one of said plates being moveable relative to the other plate by the vibrating means, said one of said plates having a side edge, and causing the sample fluid to extend up the side wall of the said one of the plates to form a concave meniscus so that upon movement of the said one of the plates, the meniscus will slip on the edge of the top plate thereby reducing the spring nature of the meniscus to reduce errors in the resulting measurement due to the spring nature of the meniscus.

Preferably the step of causing the sample fluid to extend up the side wall comprises providing said one plate with a quartz surface at least in the vicinity of the meniscus so that the fluid flows up the said side surface thereby creating the meniscus which extends up the side surface of the said one plate.

Preferably the providing step comprises vacuum depositing quartz onto the said one plate.

Preferably the quartz surface has a thickness of about 100 micrometers.

Preferably both the top plate and the bottom plate are provided with the quartz surface having the thickness of about 100 micrometers.

Preferably the said one of the plates comprises the top plate.

Brief Description of the Drawings

A preferred embodiment of the invention will be described, by way of example, with reference to the accompanying drawings, in which:

Figure 1 is a block diagram of a rheometer according to the preferred embodiment;

Figure 2 is a graph showing the sweep function of the preferred embodiment;

Figure 3 is an enlarged view of the plates of a rheometer according to the prior art; and

Figure 4 is a view similar to Figure 3, but of the preferred embodiment of the present invention.

With reference to Figure 1, a rheometer according to the preferred embodiment of the invention is shown which has a driver 10 which is preferably formed from a terfenite material and electromagnets for supplying a magnetic field to the terfenite material. Most preferably the terfenite material is in the form of rods which, when the magnetic field is applied, grow in the magnetic field. Thus, by applying an alternating magnetic field to the terfenite rods, the movement of the terfenite rods causes a vibration which creates an alternating movement of the driver 10. Such drivers are known per se and therefore need not be defined in further detail.

The driver 10 has a connecting rod 12 formed from metal which can vibrate with the driver 10, and the rod 12 is connected to top plate 14 of a sample support station 16. The driver 10 may be supported by support 18 to a casing 20 or housing of the rheometer.

The sample station 16 includes a bottom plate 22 and a space 24 is provided between the top plate 14 and the bottom plate 22 in which a sample S can be located. The bottom plate 22 is connected to a load cell 26 by a rod

28.

The top plate 14 and bottom plate 22 are preferably circular, and the top plate has a diameter a which is smaller than the bottom plate 22.

The connecting rod 12 has an abutment 30 which moves with the connecting rod 12 and a displacement transducer 32 is associated with the abutment 30 for measuring the displacement of the abutment 30 and therefore the displacement or vibration of the driver 10 and sample 12, and therefore also of the top plate 14.

Thus, the displacement transducer 32 produces a displacement signal h(t) which is indicative of the alternating movement of the top plate 14 as driven by the driver 10.

The load cell 26 produces a force signal F(t) which is indicative of the force supplied by the sample S to the bottom plate 22 upon vibrating movement of the top plate 14.

The displacement signal h(t) and the force signal F(t) are provided to processor 50. The force signal F(t) may be amplified by amplifier 52 before application to the processor 50. The processor 50 includes an analogue to digital converter 54 for converting the signal h(t) into a digital signal, and an analogue to digital converter 56 for converting the signal F(t) to a digital signal. The digital signals from the converters 54 and 56 are provided to a processing section 58 which determines the fourier transform of the signal h(t) which is expressed $H(\omega)$ and the fourier transform of the signal F(t) which is expressed as $F(\omega)$. The processor 58 also determines the ratio of the fourier transforms $F(\omega)/H(\omega)$.

The processor 50 also includes a processing section 60 for determining the complex modulus $G^*(\omega)$ of the sample which is given by the equation:

$$G^*(\omega) = h^3/3\pi a^4 \times F(\omega)/H(\omega)$$

In the preferred embodiment of the invention the displacement signal h(t) is also fed back to differential amplifier 70 and the differential amplifier also receives a frequency sweep signal from signal generator 80. The differential amplifier 70 outputs the difference of the signals $\dot{h}(t)$ and the sweep signal from the generator 80 to power amplifier 51 and then to the driver 10 so as to actuate the driver 10 and drive the driver 10 to produce the vibrating or alternating movement of the driver 10 which is imparted to the plate 14.

Figure 2 is a representative trace of the frequency sweep signal which is supplied by the generator 80 to the driver 10. The signal supplied by the generator 80 is a frequency sweep or chirp function which has a monotonic group delay function with a maximum value less than the acquisition period. Thus, the harmonics from non-linearities (ie. distortions) are distributed in a well-defined way rather than randomly across the frequency spectrum, and therefore can be corrected for in the data processing within the processor 50 so as to improve the measurement results of the complex viscosity of the sample S. This form of signal applied to the driver 10 also results in the effect of intermittent external noise sources being confined to the frequencies during which they occur, rather than being spread across the entire spectrum of the signal.

This form of signal also enables small crest factors (close to 3dB if using a flat amplitude envelope in the time domain). This is about the best practical crest

factor which can be obtained, and is therefore only 3 decibels above the theoretical best case, which cannot be achieved because of bandwidth limits. Thus, this gives the highest signal to noise ratio in terms of A/D resolution and noise generated by the sampling electronics of the displacement transducer 32 and the load cell 26.

This form of signal also provides the ability to prescribe the amplitude envelope in the time domain, and therefore enables the maximum strain rates to be constrained to within the linear region of the sample S. Furthermore, fading of the start and end points to zero amplitude is also possible, and this results in no unexpected transitions in the signal being injected into the sample, and so the sample integrity is preserved. This form of signal also provides the ability to prescribe the spectral content envelope in the frequency domain, such as flat, linear and log envelopes. This enables the rheologist to control how much energy is input into the sample at different frequencies, and also enables a signal to noise ratio of the stress measurement to be equalised, since typical samples do not have a flat transfer function.

Thus, the use of the generator 80 which produces the sweep function described above therefore provides more accurate results and better control over the analysis of samples in order to determine the complex viscosity of the sample.

Figure 3 is a diagram of the sample station 16 in which the top plates 14 and 22 of a prior art system is shown. Typically the plates 14 and 22 are formed from stainless steel and the sample S which is constrained between the plates 14 and 22 forms a meniscus 81 which extends from the lower surface 14a of the top plate to the upper surface 22a of the bottom plate. This meniscus 81 effectively acts like a spring which provides a resistance to the vibrating movement which is imparted to the sample

S by the top plate 14. This is because the meniscus 81 is constrained between the surfaces 14a and 22a and therefore must be compressed and decompressed as the plate 14a moves. This tendency of the meniscus to act like a spring therefore tends to alter the nature of the alternating flow of the sample fluid S which is created, thereby introducing errors into the measurements which are obtained.

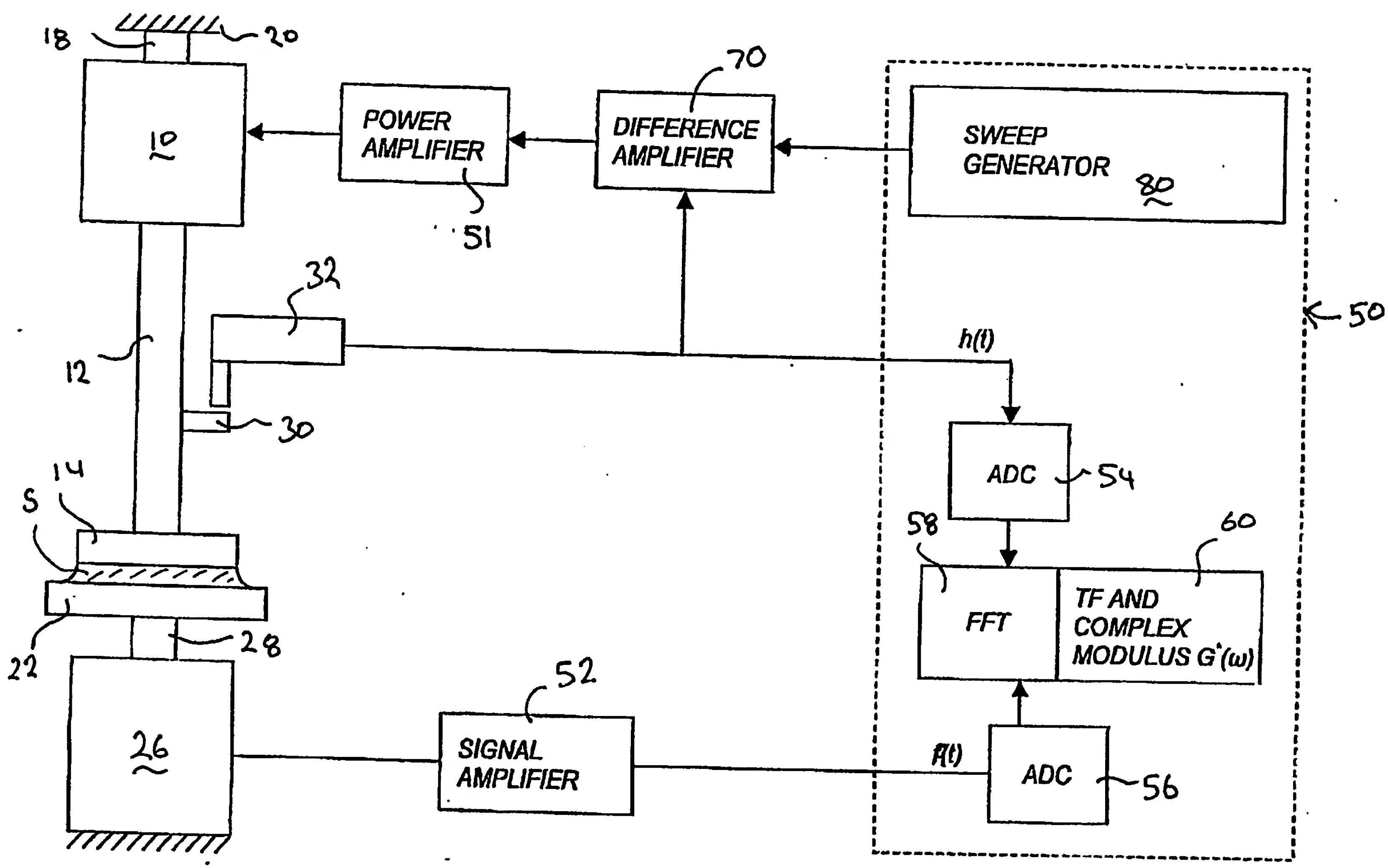
The preferred embodiment of the invention overcomes this problem by providing the plates 16 and 22 with a surface which is formed from quartz, as shown by the surface layer 83 provided on the plates 16 and 22. The surface layer 83 is preferably formed from vacuum deposition and is about 100 micrometers thick. The result of forming the quartz layer is that the meniscus 81, as shown in Figure 4, is not constrained between the surfaces 14a and 22a, but will tend to creep up the side edge 87 of the plate 16. In other words, the sample S overfills the space between the plates 16 and 22, and the meniscus 81 will slip on the side edge 87 as the plate 16 vibrates. Thus, the springing action is not produced which would cause a slight resistance to the fluid flow of the material and, according to the preferred embodiment, results are therefore improved.

The plates 16 and 22 are still formed from stainless steel but are provided with the quartz layer 83 and therefore this provides a relatively cheap sample station S, and therefore plates which are easy to clean and less porous than plain hardened stainless steel.

The meniscus 81 forms a concave shape as shown in Figure 4, and therefore a very small contact angle α between the meniscus 81 and the side edge 87. However, the meniscus 81, as is shown in Figure 5, may be concave.

By coating the plates 16 and 22 with the quartz layer 83, the aqueous samples S will therefore take up a very small contact angle, and therefore the configuration or shape as shown in Figure 4, which will not exhibit the spring nature of the meniscus and allow good slippage between the plate 16 and the fluid S. Thus, the nature of the flow of the material caused by the vibrating movement of the plate 16 is not impaired by the meniscus and therefore the signals h(t) and F(t) are more indicative of the actual nature of the sample and therefore much more accurate results are obtained.

Since modifications within the spirit and scope of the invention may readily be effected by persons skilled within the art, it is to be understood that this invention is not limited to the particular embodiment described by way of example hereinabove.



18
20
10
POWER AMPLIFIER
51
DIFFERENCE AMPLIFIER
70
SWEEP GENERATOR
80
50
32
12
30
h(t)
ADC
54
14
S
58
60
FFT
TF AND COMPLEX MODULUS G*(ω)
22
28
52
26
SIGNAL AMPLIFIER
f(t)
ADC
56

Fig 1

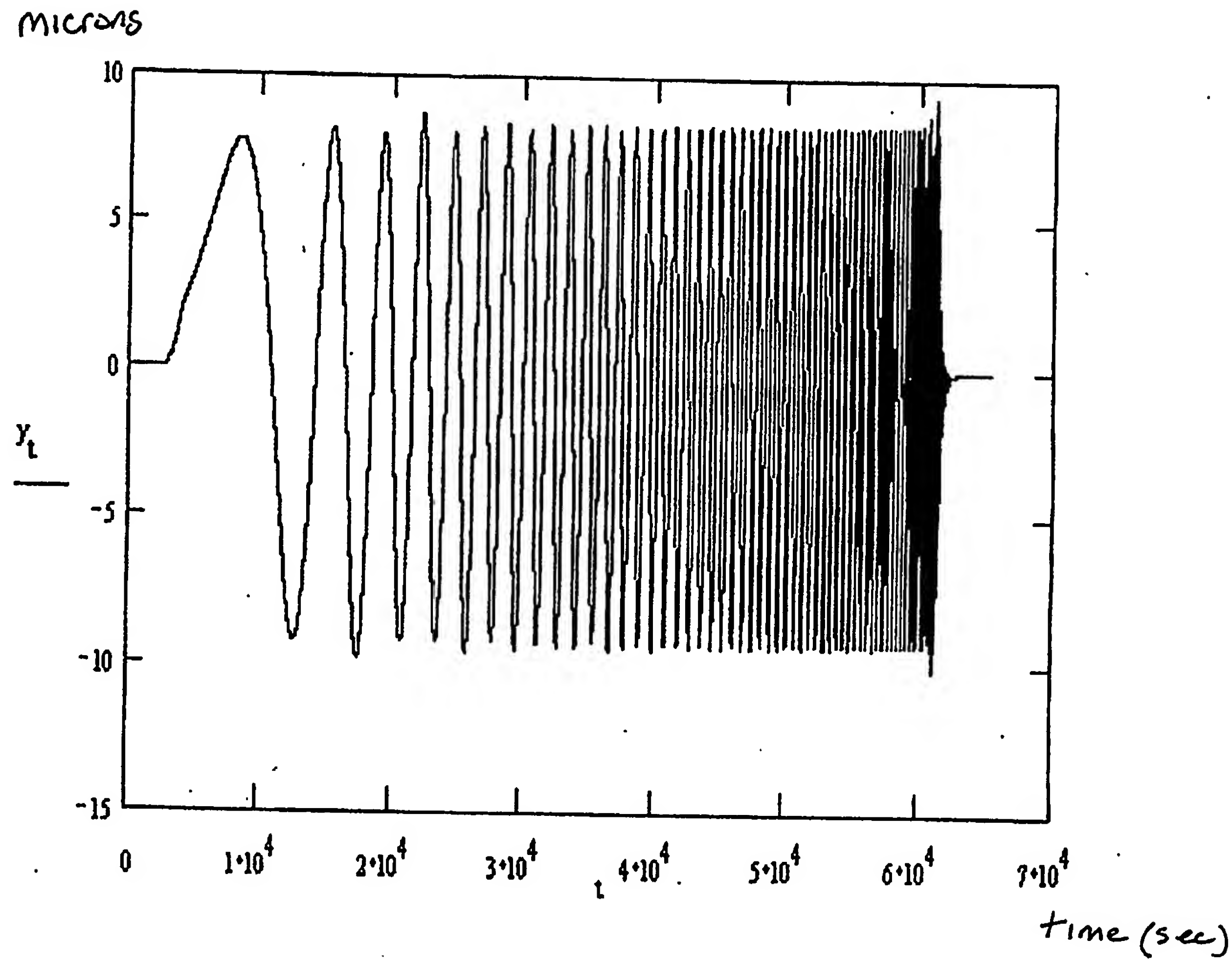
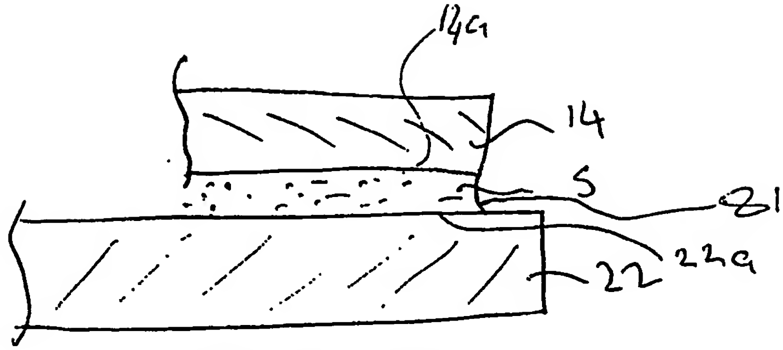

microns
10
5
0
y_t
-5
-10
-15
0
$1 \cdot 10^4$
$2 \cdot 10^4$
$3 \cdot 10^4$
t
$4 \cdot 10^4$
$5 \cdot 10^4$
$6 \cdot 10^4$
$7 \cdot 10^4$
time (sec)

Fig 2



PRIOR ART

Fig 3

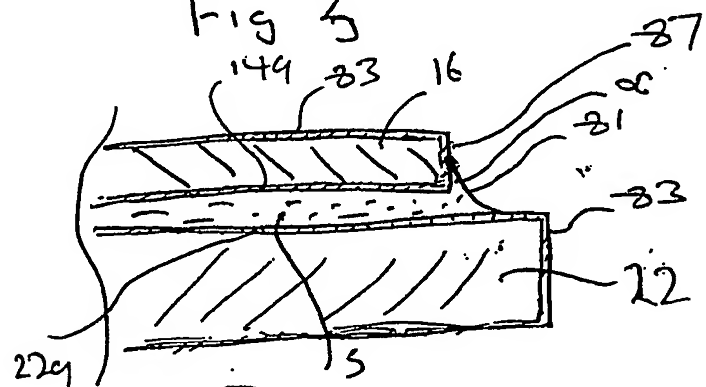


Fig 4

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